Designation: ~~D5002 – 19~~ D5002 – 22

Standard Test Method for Density, Relative Density, and API Gravity of Crude Oils by Digital Density Analyzer[1]

This standard is issued under the fixed designation D5002; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the determination of the density, relative density, and API gravity of crude oils that may be handled in a normal fashion as liquids at test temperatures between 15 °C and 35 °C utilizing either manual or automated sample injection equipment. This test method applies to crude oils with high vapor pressures provided appropriate precautions are taken to prevent vapor loss during transfer of the sample to the density analyzer.

1.2 This test method was evaluated in interlaboratory study testing using crude oils in the 0.75 g/mL to 0.95 g/mL range. Lighter crude oil may require special handling to prevent vapor losses.

1.3 The values stated in SI units are to be regarded as standard. Other units of measurement are included in this standard. The accepted units of measurement of density are grams per millilitre and kilograms per cubic metre.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 7.4, 7.5, and 7.6.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer (Withdrawn 1993)[3]

D1193 Specification for Reagent Water

D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

D1250 Guide for the Use of the Joint API and ASTM Adjunct for Temperature and Pressure Volume Correction Factors for Generalized Crude Oils, Refined Products, and Lubricating Oils: API MPMS Chapter 11.1

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0D on Physical and Chemical Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

***A Summary of Changes section appears at the end of this standard**

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (Withdrawn 2020)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *density, n*—mass per unit volume at a specified temperature.

3.1.1.1 *Discussion*—
The SI unit of density is kg/m^3; the unit of measure g/cm^3 is commonly used in industry. Less preferred units, for example, kg/L or g/mL, are still in use.

3.1.2 *relative density, n*—the ratio of the density of a material at a stated temperature to the density of water at a stated temperature.

3.1.2.1 *Discussion*—
Relative density is also commonly known as specific gravity. Commonly used stated temperatures are 20 °C/20 °C, 15 °C/15 °C, 20 °C/4 °C and 60 °F/60 °F. "Relative density" was historically known as the deprecated term "specific gravity".

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *API gravity, n*—a special function of relative density 60 °F/60 °F, represented by:

$$°\text{API} = \frac{141.5}{\text{relative density}} - 131.5 \quad (1)$$

3.2.1.1 *Discussion*—
No statement of reference temperature is required since 60 °F is included in the definition.

3.2.2 *test specimen, n*—the volume of the sample aliquot residing in the U-tube during the measurement cycle.

3.2.2.1 *Discussion*—
Sample material residing in filling nozzles, tubing and valve manifolds is not considered "Test Specimen." A test specimen may be measured only once.

4. Summary of Test Method

4.1 Approximately 1 mL to 2 mL of crude oil sample is introduced into an oscillating U-tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with adjustment data to determine the density, relative density, and API gravity of the sample. Both manual and automated injection techniques are described.

5. Significance and Use

5.1 Density is a fundamental physical property that may be used in conjunction with other properties to characterize the quality of crude oils.

5.2 The density or relative density of crude oils is used for the conversion of measured volumes to volumes at the standard temperatures of 15 °C or 60 °F and for the conversion of crude mass measurements into volume units.

5.3 The application of the density result obtained from this test method, for fiscal or custody transfer accounting calculations, may require measurements of the water and sediment contents obtained on similar specimens of the crude oil parcel.

6. Apparatus

6.1 *Digital Density Analyzer*—A digital analyzer consisting of a U-shaped, oscillating tube, U-tube, and a system for electronic excitation, frequency counting, and display. The analyzer shall accommodate the accurate measurement of the sample temperature during measurement or shall control the sample temperature. The instrument shall be capable of meeting the precision requirements described in 6.1 of Test Method D4052.

6.2 *Syringes,* at least 2 mL in volume with a tip or an adapter tip that will fit the inlet of the density analyzer.

6.3 *Flow-Through or Pressure Adapter,* for use as an alternative means of introducing the sample into the density meter.

6.4 *Autosampler,* required for use in automated injection analyses. The autosampler shall be designed to ensure the integrity of the test specimen prior to and during the analysis and be equipped to transfer a representative portion of test specimen to the digital density analyzer.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D1193 or better.

7.3 *Water,* reagent water, freshly boiled, to remove dissolved gasses, for use as a primary calibration standard. (**Warning—** Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.)

7.4 *Acetone,* for flushing and drying the sample tube. (**Warning—**Extremely flammable.)

7.5 *Petroleum Naphtha,* for flushing viscous petroleum samples from the sample tube. (**Warning—**Extremely flammable.)

NOTE 1—Suitable solvent naphthas are marketed under various designations such as "petroleum ether," "ligroine," or "precipitation naphtha."

7.6 *n-Nonane, n-tridecane or cyclohexane,* 99 % purity or better, or similar pure material for which the density is known precisely from literature references or by direct determination in accordance with Test Method D941 or D1217. (**Warning—**Extremely flammable.)

8. Sampling, Test Specimens, and Test Units

8.1 Sampling is defined as all the steps required to obtain an aliquot of the contents of any pipe, tank or other system, and to place the sample into the laboratory test container. The laboratory test container and sample volume shall be of sufficient dimensions to allow mixing as described in 8.3.1. Mixing is required to obtain a homogeneous sample for analysis.

8.2 *Laboratory Sample*—Use only representative samples obtained as specified in Practices D4057 or D4177 for this test method.

8.3 *Test Specimen*—A portion or volume of sample obtained from the laboratory sample and delivered to the density analyzer U-tube. Obtain the test specimen as follows:

8.3.1 Mix the sample of crude oil to homogenize any sediment and water present. The mixing may be accomplished as described in Practice D4177 or Test Method D4377. Mixing at room temperature in an open container can result in the loss of light ends, so mixing in closed, pressurized containers or at sub-ambient temperatures is recommended.

8.3.2 Draw the test specimen from a properly mixed laboratory sample using an appropriate syringe. Alternatively, if the proper density analyzer attachments and connecting tubes are used then the test specimen may be delivered directly to the analyzer's

[4] ~~*Reagent Chemicals, American Chemical Society Specifications,*~~*ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials,* American Chemical Society, Washington, DC. For ~~Suggestions~~suggestions on the testing of reagents not listed by the American Chemical Society, see ~~*Annual*~~*Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Density of Water[A]

NOTE 1—Several metrological entities have issued water density tables and alternative water density data is referenced in publications external to ASTM and this test method. Using water density data from an alternative recognized source does not pose a compliance issue with this test method as the variation in the data typically is limited to the sixth decimal place.

Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature,° C	Density, g/mL
0.01	0.999844	21.0	0.997996	40.0	0.992216
3.0	0.999967	22.0	0.997773	45.0	0.990213
4.0	0.999975	23.0	0.997541	50.0	0.988035
5.0	0.999967	24.0	0.997299	55.0	0.985693
10.0	0.999703	25.0	0.997048	60.0	0.983196
15.0	0.999103	26.0	0.996786	65.0	0.980551
15.56	0.999016	27.0	0.996516	70.0	0.977765
16.0	0.998946	28.0	0.996236	75.0	0.974843
17.0	0.998778	29.0	0.995947	80.0	0.971790
18.0	0.998599	30.0	0.995650	85.0	0.968611
19.0	0.998408	35.0	0.994033	90.0	0.965310
20.0	0.998207	37.78	0.993046	99.9	0.958421

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Lemmon, E. W., McLinden, M. O., and Friend, D. G., "Thermophysical Properties of Fluid Systems,"*NIST Chemistry WebBook*, NIST Standard Reference Database No. 68, Eds. P.J. Linstrom and W.G. Mallard, National Institute of Standards and Technology, Gaithersburg, MD, http://webbook.nist.gov. (retrieved July 24, 2013).

U-tube from the mixing container. For automated injections, it is necessary to first transfer a portion of sample by appropriate means from a properly mixed laboratory sample to the autosampler vials and take the necessary steps to ensure the integrity of the test specimen prior to and during the analysis. Sample vials for the autosampler shall be sealed immediately after filling up to 80 % ± 5 % and shall be kept closed until the auto sampler transfers the test specimen into the U-tube.

9. Preparation of Apparatus

9.1 Set up the density analyzer following the manufacturer's instructions. Set the internal temperature control so that the desired test temperature is established and maintained in the U-tube compartment of the analyzer. Verify the calibration of the instrument at the same temperature at which the density or relative density of the sample is to be measured.

10. Verification and Adjustment

10.1 As a minimum requirement, calibration verification of the instrument is required when first set up and whenever the test temperature is changed. Thereafter, conduct calibration verification at least weekly during routine operation or more frequently as may be dictated by the nature of the crude oils being measured. Whenever the apparatus fails a calibration verification without discernible cause, the apparatus shall be adjusted.

10.1.1 For non-calculating density meters, see Appendix X1 for adjustment procedure.

10.2 The adjustment routine for digital density meters involves using a minimum of two reference media. Typically, this will be air and freshly boiled reagent water under atmospheric conditions. (**Warning—**Handling water at boiling or near boiling temperature may present a safety hazard. Wear appropriate personal protective equipment.) Other materials such as n-nonane, n-tridecane, cyclohexane, and n-hexadecane (for high temperature applications) may also be used as appropriate adjustment materials, provided the reference materials have density values that are certified and traceable to national standards.

10.3 Follow the manufacturer's instructions for the proper adjustment of the apparatus. If the apparatus is adjusted using air and reagent water, observe the proper entries of air and water density values (see Table 1).

10.3.1 The density of air varies with pressure and relative humidity. Therefore, it is important that the dewpoint of ambient air is below the adjustment temperature of the instrument as to avoid condensation of water in the U-tube. This may be achieved by flushing ambient air through a desiccant container and into the U-tube.

10.3.2 The density of air varies with ambient pressure as a consequence of site elevation and atmospheric changes in pressure. The air density may be calculated using this formula:

$$\rho_{air} = 0.001293[273.15/T][P/101.325]\ g/mL \quad (2)$$

where:

ρ_{air} = density of air,
T = temperature, K, and
P = site atmospheric pressure at the time of adjustment, kPa.

NOTE 2—P should preferably be determined by direct measurement of the barometric pressure at the site of calibration. If direct measurement is not available, and common sources providing weather data are consulted, the pressure reported is typically corrected to Sea Level, P_{SL}. Therefore, such pressure data shall be corrected back to site pressure, P. For correction of P_{SL} to P:

$$P = P_{SL2} - [\text{Site Elevation (meters)}/82.3] \quad (3)$$

NOTE 3—In the International Standard Atmosphere, ISA, the pressure drops 1 kPa per 82.3 m of elevation

10.3.3 The water density values are given in Table 1. Water density values are considered constant with respect to pressure in the range of normally occurring atmospheric pressure.

NOTE 4—The need for a change in adjustment is generally attributable to deposits in the sample tube that are not removed by the routine flushing procedure. Although this condition may be compensated for by adjusting the apparatus, it is good practice to clean the tube with warm chromic acid solution (**Warning—**Causes severe burns. A recognized carcinogen.) whenever a major adjustment is required. Chromic acid solution is the most effective cleaning agent; however, surfactant-type cleaning fluids have also been used successfully.

11. Procedure

11.1 Introduce a minimum of 1 mL to 2 mL of crude oil into the clean, dry, U-tube of the instrument using a suitable syringe. Leave the syringe in place.

11.1.1 Ensure that the U-tube is properly filled and that no gas bubbles are present. The sample shall be homogeneous and free of even the smallest gas bubbles. Check the integrity of the filled sample by using optical or physical methods to verify absence of gas bubbles. If gas bubbles are detected, empty and refill the sample tube, and recheck for gas bubbles.

11.1.2 Allow the sample to equilibrate to the test temperature before proceeding to evaluate the test sample for the presence of unseen air or gas bubbles.

11.1.3 For dark crude oil samples the observation of air or gas bubbles in the U-tube is very difficult. The presence of bubbles may often be detected, however, by observing the fluctuations of the digital display of the density value. Air or gas bubbles cause large random variations in the third and fourth significant figures for density reading. When bubbles are absent and the sample is at equilibrium with the test temperature, the displayed values are stable, do not drift, and show only small variations of the order of ±1 to 2 units in the last significant figure. If stable values are not observed after a few minutes, then repeat the injection of a new test specimen into the U-tube.

NOTE 5—When viscous liquids are being measured, a stable reading may be achieved even when air or gas bubbles are present. Careful injection of fresh sample will often eliminate bubbles. Since bubbles contribute to lower density readings, an observed increase in the density of the liquid after injection of fresh test specimen tends to suggest that bubbles were previously present.

11.1.4 After the instrument displays a steady reading to four significant figures for density, relative density, and two significant figures for API gravity, indicating that temperature equilibrium has been reached, record the density, relative density, and API gravity.

11.1.5 Flush and dry the U-tube as described in X1.1.1 and check the calibration as described in X1.2.1 prior to introducing another sample.

11.2 *Automated Injection:*

11.2.1 The use of an autosampler (see 6.4) is required when analyzing samples by automated injection. Follow manufacturer's instructions for ensuring the integrity of the test specimen prior to analysis, as well as for transferring a representative test specimen into the U-tube for analysis.

11.2.2 When using an autosampler for samples expected or known to contain high quantities of volatile components, use two separate test specimens per sample, in order that errors due to potential sample handling of volatile materials and potential gas bubble formation may be detected and the system performance monitored. For all other samples, a single determination using an autosampler is sufficient.

11.2.2.1 If the lab decides to perform a second automated injection determination for a given sample, the differences between each determination should not exceed a determinability criterion determined by a series of tests on a representative crude sample and which assures that the repeatability performance of 14.1.1, Table 2 is met. Averaged results meeting the necessary acceptance criteria are to be used for reporting purposes.

11.2.2.2 If the two determinations fall outside this acceptance criteria, both determinations are to be discarded and 11.2.2 shall be repeated until the acceptance criteria identified in the previous sentence is satisfied. In cases where the acceptance criteria is not initially satisfied, the lab may need to investigate and take corrective actions before proceeding with subsequent analyses.

11.3 Record the density, relative density, and API Gravity results, or a combination thereof, determined by the analyses as appropriate, such as by using the instrument print out of results to meet the recording requirements.

12. Calculation

12.1 *Calculating Density Analyzers*—The recorded value is the final result, expressed either as density in g/mL, kg/m^3 or as relative density or API gravity. Note that kg/m^3 = 1000 × g⁄mL.

12.2 If it is necessary to convert a result obtained using the density meter to API Gravity, or a density or relative density at another temperature, Guide D1250 may be used only if the glass expansion factor has been excluded.

13. Report

13.1 In reporting density, give the test temperature and the units, (for example: density at 20 °C = 0.8765 g⁄mL or 876.5 kg⁄m^3 (in vacuo)). Report the final result to four significant figures and reference this test method.

13.2 In reporting relative density, give both the test temperature and the reference temperature, but no units, (for example: relative density, 15/15 °C = x.xxxx). Report the final result to four significant figures.

14. Precision and Bias[5,6]

14.1 The precision of this test method as obtained by statistical examination of interlaboratory test results at test temperatures of 15 °C and 20 °C is as follows:

14.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the following value only in 1 case in 20 (see Table 2):

range	repeatability
0.75 to 0.95	0.00105*X*

where:

X = sample mean.

14.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20 (see Table 2):

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1257. Contact ASTM Customer Service at service@astm.org.

[6] Biased results for high viscosity samples (>ca. 100 mPa-s dynamic viscosity) has been reported in the literature. For additional information, consult the *Journal of Physical Chemistry*, Vol 84, 1980, pp. 158–162 and the *Journal of the Chemical Society Faraday Translation*, Vol 86_(1), 1990, pp. 145–149.

TABLE 2 Precision Values

Density	Repeatability Density	Repeatability API Gravity	Reproducibility Density	Reproducibility API Gravity
~~0.70~~	~~0.0007~~	~~0.10~~	~~0.0029~~	~~0.41~~
~~0.75~~	~~0.0008~~	~~0.11~~	~~0.0031~~	~~0.44~~
~~0.80~~	~~0.0008~~	~~0.11~~	~~0.0033~~	~~0.47~~
~~0.85~~	~~0.0009~~	~~0.13~~	~~0.0035~~	~~0.50~~
~~0.90~~	~~0.0009~~	~~0.13~~	~~0.0037~~	~~0.53~~
~~0.95~~	~~0.0010~~	~~0.14~~	~~0.0039~~	~~0.55~~

TABLE 2 Precision Values

Density	Repeatability Density	Repeatability API Gravity	Reproducibility Density	Reproducibility API Gravity
0.70	0.0007	0.21	0.0029	0.83
0.75	0.0008	0.20	0.0031	0.77
0.80	0.0008	0.19	0.0033	0.73
0.85	0.0009	0.17	0.0035	0.68
0.90	0.0009	0.16	0.0037	0.64
0.95	0.0010	0.16	0.0039	0.61

range	reproducibility
0.75 to 0.95	0.00412*X*

where:

X = sample mean.

NOTE 6—Table 2 values must not be used to qualify density meter systems or in certification of results. For such purpose, only use stated values for repeatability and reproducibility.

14.2 *Bias*—After suggestions of its existence from literature,[7] a study has been performed which has confirmed the presence of a bias between known density values for reference materials and from values determined according to this test method on the same reference materials. The matrix for this bias study was comprised of 15 participants, each analyzing four reference oils with certified density values, established by the Netherlands Meet Instituut (NMI), by pycnometry, covering densities in the range of 747 kg/m^3 to 927 kg/m^3 at 20 °C, with viscosities between 1 mPa.s and 5000 mPa.s (also at 20 °C). Users should, therefore, be aware that results obtained by this test method may be biased by as much as 0.6 kg/m^3 (0.0006 g/mL).[8]

15. Keywords

15.1 API gravity; crude oils; density; digital density analyzer; relative density

APPENDIXES

(Nonmandatory Information)

X1. ADJUSTMENT AND CALIBRATION PROCEDURE FOR NON-CALCULATING DENSITY METERS

X1.1 Initial calibration, or calibration after a change in test temperature, necessitates calculation of the values of the Constants A and B from the periods of oscillation, (*T*), observed when the sample cell contains certified reference liquids such as air and freshly boiled reagent water. (See Warning note in 7.3.) Other calibrating materials such as n-nonane, n-tridecane, cyclohexane, and n-hexadecane (for high temperature applications) may also be used as appropriate.

X1.1.1 While monitoring the oscillator period, *T*, flush the sample tube with petroleum naphtha, followed with an acetone flush and dry with dry air. Continue drying until the display exhibits a steady reading. In cases where saline components can be deposited in the cell, flush with distilled water followed by acetone and dry air. Contaminated or humid air can affect the calibration. When

[7] Fitzgerald, H. and D., "An Assessment of Laboratory Density Meters," *Petroleum Review*, November 1992, pp. 544–549.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1387. Contact ASTM Customer Service at service@astm.org.